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MICROSTRUCTURE ANALYSIS OF BORON NITRIDE

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14. ABSTRACT

Transmission electron microscopy and electron diffraction technique revealed boron nitride nanotube (BNNT) and nanoparticles in thin film baron nitride prepared by chemical vapor deposition method. Electron micrographs show single walled nanotubes containing these nanoparticles. Electron diffraction pattern confirms this boron nitride has a hexagonal crystal structure.

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INTRODUCTION

Boron nitride nanotubes (BNNT) are equally acceptable for application as carbon nanotubes (CNT). The BNNT is always semiconducting (above 4ev gap); whereas, the CNT could be metallic or semiconducting. Properties of boron nanotubes in contrast to CNT have already been reported (ref. 1). Chemical vapor deposition (ref. 2) has previously synthesized hexagonal boron nitride (h-BN). The present BN thin film sample was prepared at the U.S. Army Armaments Research, Development and Engineering Center (ARDEC), Picatinny Arsenal, New Jersey by the Energetic and Warhead Division. Iron boride (FeB) powder was nitrided in NH_3 at 425°C for 24 hrs producing non-crystalline h-BN and Fe_3N. After annealing for 1 hr at 1050°C, the iron-containing phase was removed with dilute mineral acid leaving h-BN. The purpose of this report is to describe the microstructural characterization of this h-BN thin film using a Philips 420 transmission electron microscope (TEM). These samples show a mixture of BNNT and nanoparticles (NPs). The selected area electron diffraction (SAED) pattern obtained from this sample shows a six-fold symmetry confirming this BN thin film has a hexagonal crystal structure and individual nanotubes are well as crystalline. The BN is chemically inert and has a high temperature resistance to oxidation, thus suitable for hydrogen storage and weapon systems.

SPECIMEN PREPARATION

A thin film of h-BN was supported on a 200-mesh holey carbon coated copper grid. This sample was provided to the ARDEC electron microscope laboratory for microstructure characterization. A single tilt specimen holder was used for this sample and inserted in the TEM.

RESULTS AND DISCUSSION

All electron micrographs were taken at 100 kv electron beam emitting from a tungsten filament. Smallest condenser aperture and beam spot sizes with longer exposure time were used for recording electron diffraction patterns. A digital close circuit Gatan camera attached to a computer was used to record the digital pictures. Figure 1 shows a bright field electron micrograph from BN thin film supported by holey carbon grids. Figure 2 shows another electron micrograph taken from a different area of the specimen at higher magnification revealing BNNT indicated by 't'. This micrograph also shows NPs as black dots. Figure 3, at much higher magnification, reveals single walled nanotubes indicated by 't' having different widths and length. Black spherical spots are due to NPs. The TEM of a single to six-walled BNNT have already been reported (ref. 1). A nanotube can be described as a long thin strip, cut of a single atomic plane of material, rolled to form a cylinder with a diameter of nanometer scale and length on the order of microns (ref. 3).

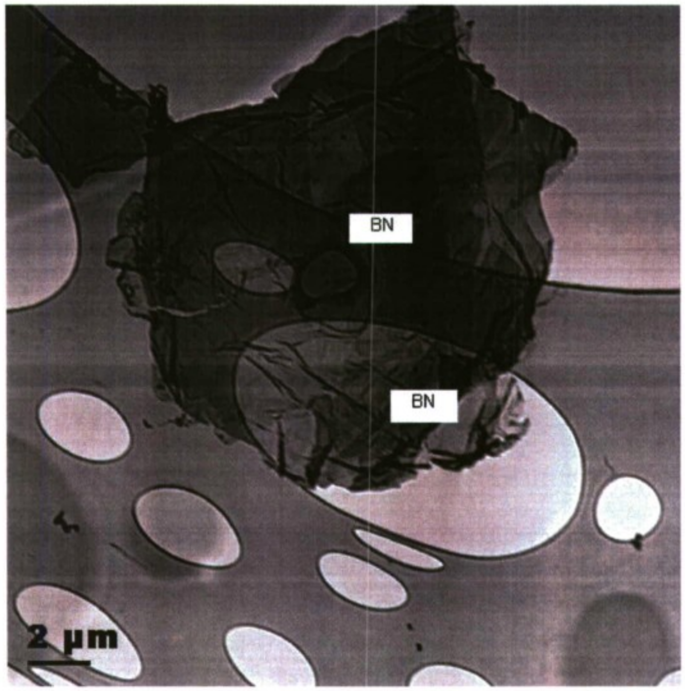

Figure 1
Bright field picture of BN showing specimens supported on the holey carbon grid

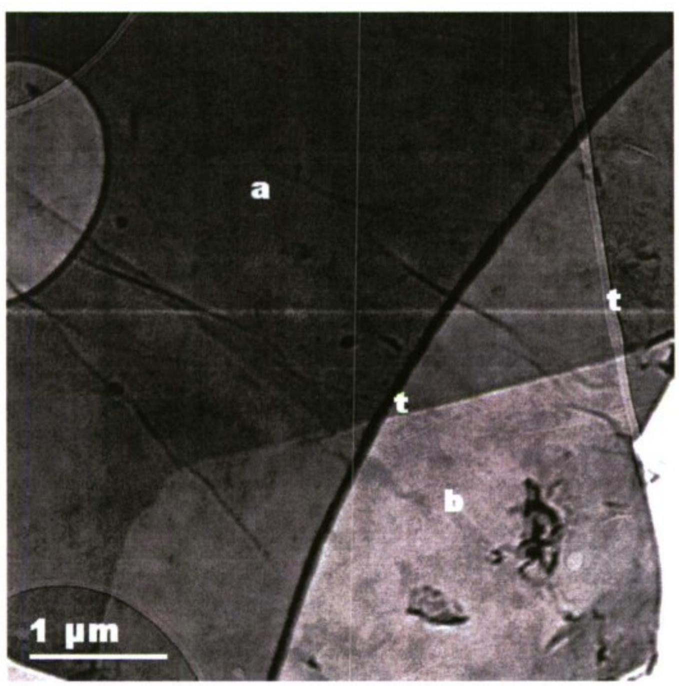

Figure 2
Microstructure of BN showing nanotubes (t) at low magnification

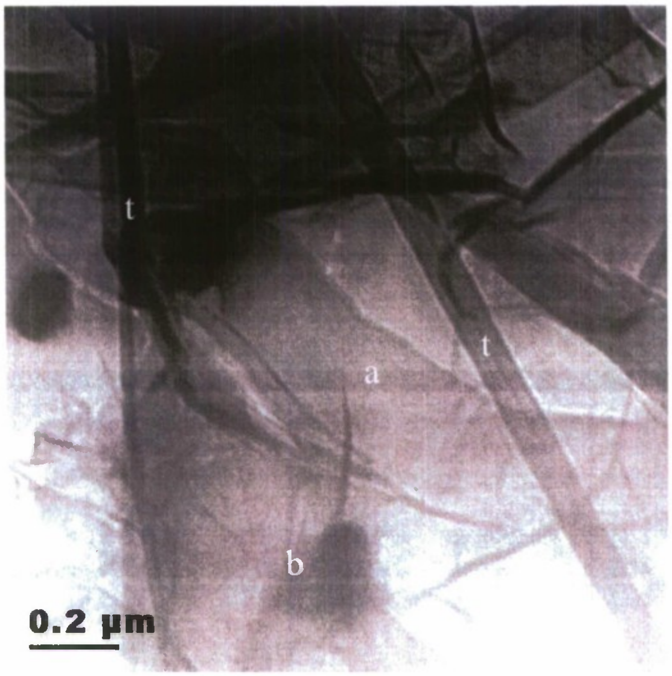

Figure 3
TEM taken from different areas of BN sample at higher magnification

Figure 4 shows an electron diffraction pattern taken from an area of the samples with BNNT. This diffraction pattern has a first order and second order six fold symmetry confirming this BN film has h-BN. Those diffraction spots are due to reciprocal lattice points. Actually, these single spots are split into double spots indicating double diffraction reciprocal lattice points. Unfortunately, figure 4 did not resolve these double spots though it was observed on the digital image on the computer screen. This double diffraction pattern originated from the double layered BNNT, which could not be seen on the electron micrograph due to poor resolution. Figure 5 shows a bundle of randomly oriented BNNTs with different sizes as indicated by ‘nt’ and ‘d’. Figure 6 shows two nanotubes P1 and P2 adjacent two each other and passing through the wall W of the thin folded BN film. Figure 7 reveals two narrow nanotubes ‘nt’ and a wide nanotube ‘a’ together with a network ‘d’ consisting of ledges before the growth to form a nanotube.

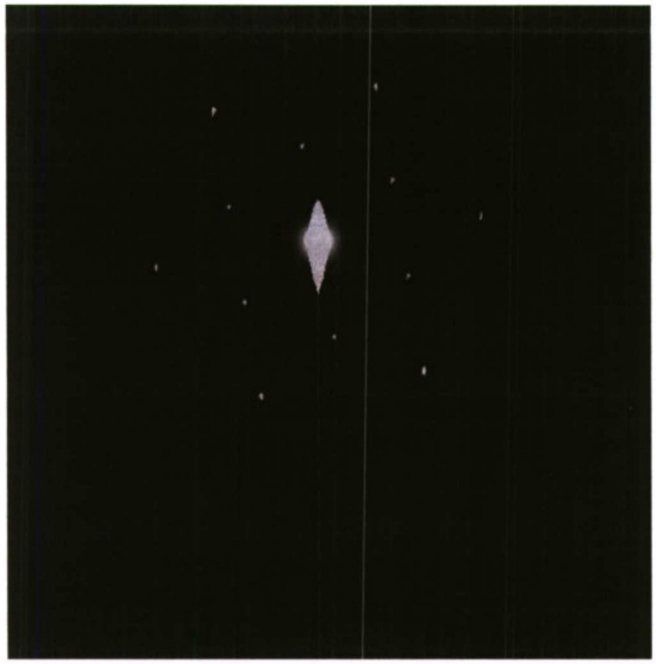

Figure 4
SAED from area shown in figure 3
(The six-fold symmetry of the diffraction pattern confirmed that the BN has a hexagonal structure)

Figure 5
TEM at high magnification showing a bundle of nanotubes of different dimensions

Figure 6
Bright field TEM of BN at higher magnification from another area

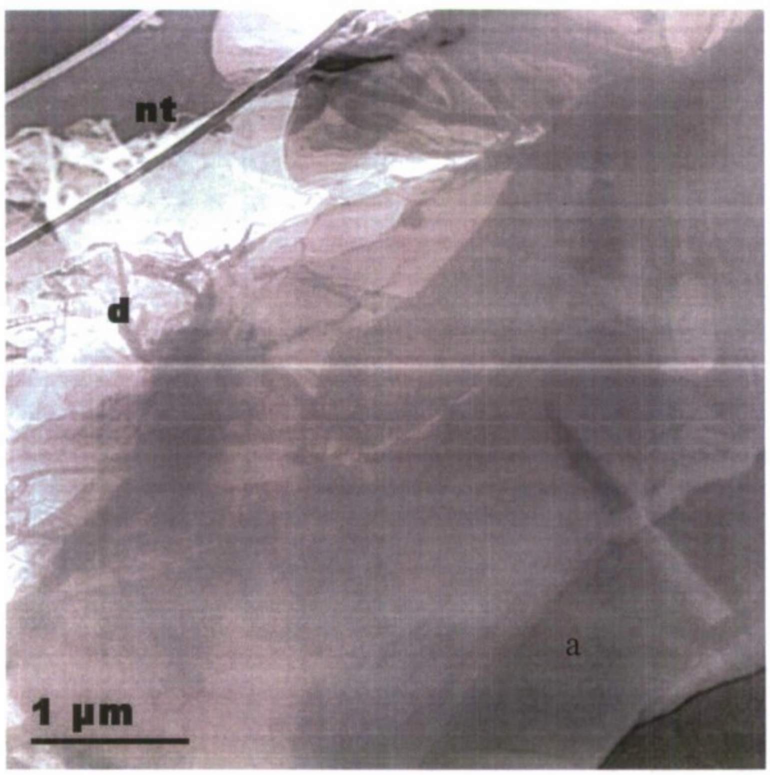

Figure 7
Microstructure of BN bounded by nanotubes (nt)

REFERENCES

1. Ishigami, Masa; Aloni, Shaul; and Zetti, A.; http://proceedings.aip.org/proceedingscpcr.jsp

2. Warner, T.E. and Fray, D.J.; Journal of Materials Science, 35; 2000.

3. Chopra, Nasreen G. and Zetti, A.; A New Direction: Nanotubes; pages: 189-197.

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